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EPA **Project Summary**

Round-Robin Testing of Methods for Collecting Dislodgeable Residues from Carpets

Christopher R. Fortune

A round-robin test was conducted using six volunteers to evaluate three dislodgeable residue methods sampling new carpets treated with a commercial pesticide formulation. Seven separate tests were performed, each using a formulation containing three target pesticides (chlorpyrifos, pyrethrin I, and piperonyl butoxide). Strict QA/QC guidelines were followed as each participant collected three replicate samples each with the polyurethane foam (PUF) roller, the California roller, and the Dow drag sled methods. Sampling precision was high for all three methods for measurements of this type. The overall results (mean %RSD, relative standard deviation, N=21) show the Dow sled with the best sampling precision (25.4% RSD), followed by the California roller (30.7%), and then the PUF roller (37.9%). Mean transfer efficiency, the ratio of the method transfer rate to the pesticide deposition rate, was highest for the California roller (5.0%), followed by the Dow sled (2.1%) and the PUF roller (1.7%). The mean transfer efficiency rates in this study were substantially higher than those reported in earlier studies of this type.

Information relating to ease of use, simplicity, time requirements, and other criteria for each of the test methods was obtained from written subjective evaluation and critique by each volunteer. A compilation of that information revealed that both the Dow sled and PUF roller methods were rated highly and equal to one another, while the California roller was rated lower. Further testing is recommended to determine the effect on transfer efficiency rates due to carpet age, type and prior cleaning or chemical treatment.

This Project Summary was developed by EPA's National Exposure Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

A recent study was conducted to test the performance of three dislodgeable pesticide residue methods on floor surfaces. One result of that study was a recommendation to conduct a round robin study of the same three methods, following strict QA/QC guidelines, using volunteers to perform the testing and provide written evaluations of the procedures. This report presents the results of that round-robin study using six volunteers.

The PUF roller, the California roller and the Dow drag sled methods were evaluated. Written standard operating procedures (SOPs) were prepared, tested and modified, where necessary, for each method prior to the onset of the testing. Volunteers were trained in the use of each procedure just prior to beginning the sampling activities. For each test a new, nylon, plush cut-pile, stain resistant carpet was treated with a commercial pesticide formulation used to control a heavy infestation of fleas. Field blank and field spike samples were collected along with the replicate samples collected using the three test methods.

Test samples, along with both field and laboratory QA/QC samples, were extracted and then analyzed by a referee laboratory using a GC/MS SIM mode procedure. The analytical results for the three target analytes, chlorpyrifos, pyrethrin I and piperonyl butoxide, were used to calculate

the method sampling precision and the transfer efficiency rate for each test procedure. Upon the conclusion of each test, the volunteer provided a written evaluation pertinent to a wide variety of specific performance characteristics for each test method used in the study.

Study Design

Six volunteers were recruited from local agricultural and chemical research organizations. Half of the participants were from large organizations and the other half were from smaller companies. An 8- x 20-ft modular trailer unit was leased for use in performing the tests. Participants were provided advanced copies of the SOPs and general instructions concerning the planned training and testing activities. On the morning of each scheduled test, the staff collected field blanks from a new 6- x 14-ft carpet test sample and then treated the carpet with the freshly prepared test formulation. A mixture of 0.5% chlorpyrifos, 0.05% pyrethrin I, and 0.5% piperonyl butoxide was sprayed on the carpet surface at a manufacturer's recommended rate of 1 gal/1600 ft^2. The carpet was divided into three equal sections for sampling layout purposes, and a 4- x 4-in. α-cellulose deposition coupon was placed in each section prior to spraying in order to provide a measure of the actual pesticide deposition rate for each test.

Shortly after arriving at our test facility, the volunteers were given a thorough demonstration and hands-on familiarization session with the three dislodgeable residue test methods. After a 4 hour drying period and complete ventilation, the testing session was initiated. A set of field spikes of each of the four sample matrices was collected in the laboratory. The volunteers then performed the replicate sampling using each test method to collect one sample in each of the three sections of the test carpet. The exposed deposition coupons were collected from each section individually at the time test samples were collected.

All samples were stored in pre-labled containers, packed on dry ice, and shipped to the laboratory by overnight express delivery service. Participants were debriefed following the testing and were asked to rate the test procedures by filling out questionnaires provided for that purpose.

The analytical laboratory services were provided by Southwest Research Institute, San Antonio, TX. Having had previous experience in studies of this kind, the laboratory followed written protocols in performing the extraction and GC/MS analysis of the test samples and laboratory QA/QC samples. The resulting data were used to determine the overall quality of the sample extraction and analysis procedures, the uniformity of pesticide applications, and the overall sampling precision and transfer efficiency of each test method under evaluation.

Test Methods

α–Cellulose Deposition Coupon

The deposition coupons used were 100-mm squares of cellulose filter paper attached to aluminum foil-covered backing sheets of the same size. Each coupon was marked by pencil line so that a 12.5-mm (0.5 in.) border on all sides was available to allow handling. Prior to extraction, the coupon border was trimmed away to leave a 75-mm square (56.25 cm^2) filter sample for analysis in the laboratory. Estimates of the deposition rate for a given sample were determined from the ratio of the mass of pesticide residue found on the coupon to its area and were usually reported in units of micrograms per square centimeter.

PUF Roller

The PUF roller dislodgeable residue sampling method used a prototype mechanical apparatus having two rear wheels, a cylindrical aluminum roller at the front, and a handle for pushing or pulling attached at the rear. Two stainless steel blocks (total weight = 3.875 kg) were attached to the center portion of the frame. A polyurethane foam (PUF) ring measuring 90-mm o.d.- x 30-mm i.d.- x 76 mm in length was fitted onto the aluminum cylinder, which was then attached to the front of the sampler assembly.

The location of the sample was marked on the test surface by placing a metal guidebar adjacent to the planned track of the device. The 100-cm sample traverse distance was marked off in 10-cm segments on the bar. A solvent-washed thin aluminum sheet was placed at the starting position to serve as a platform to hold the PUF roller before starting the test.

A sample traverse consisted of two passes, one forward and one backward, over the 100 cm pathlength. At the end of the second pass, the roller was immediately lifted off the carpet. The aluminum cylinder was then detached from the assembly and the PUF ring was recovered and stored in its container. The marks at 10 cm intervals on the guidebar were used to maintain the sampling rate at about 10 cm/s.

California Roller

The California roller method used a device resembling a large rolling pin to collect surface dislodgeable residues on a percale sheet cloth matrix. The roller consisted of a 63-cm-long PVC pipe, 13 cm in diameter, fitted with PVC endcaps having roller handles. The roller was covered with a 1-cm thick foam cover, 51 cm in length, and is filled with a quantity of small to medium-size steel ball bearings sufficient to bring the total weight of the roller to 25 lb.

The sampling medium, consisting of a precleaned 17- x 17-in. cloth cut from percale bedsheet material (50% combed cotton, 50% Fortrel® polyester, 180 thread count), was placed flat on the carpet surface and covered with a plastic sheet (e.g. medium-size plastic trash bag, 20- x 24-in.) Sampling$~as performed by moving the roller back and forth ten times at a steady rate over the sample medium (20 total passes). A special metal handle assembly was fabricated for use in these tests to permit the operator to more comfortably move the roller without exerting any downward pressure on the handles. After the final sample pass, the roller and handle assembly were removed from the test area, the plastic sheet discarded and forceps used to collect and fold the percale cloth for insertion into its sample container.

Dow Drag Sled

The Dow drag sled procedure used a 3- x 3-in. piece of 3/4-in. thick plywood as the base for a sled with a ridge contructed on the top to hold an 8-lb round downrigger weight. A screw eye was placed in the center of one edge of the block, to which a 24-in. long wire and pull handle were attached. The base and sides of the sled were covered with two layers of aluminum foil attached with staples.

The sampling media consisted of precleaned 4- x 4-in. undyed cotton denim cloth squares. A thin aluminum sheet starting platform and a guidebar were used with the Dow sled procedure. A sample traverse consisted of a single pass with the sled over a distance of 48 in. using guide marks at 4 in. intervals to achieve an approximate sampling rate of 10 cm/s.

The denim cloth sampling media was attached to the base of the sled by using plastic-headed pushpins positioned through the overlapping edges of the cloth and into the front and sides of the wooden sled. With the denim cloth securely in place, the sled was placed on the aluminum starting platform, and the 8-lb. weight was put into its position on top of the sled. The drag line was then attached to the screw eye on the front of the sled. Keeping the drag line at a low angle relative to the floor, the operator pulled the sled forward at a steady rate until the front of the

sled reached the 48-in. mark on the guidebar. The sled was then quickly lifted from the carpet surface and the denim cloth was removed from the sled, folded and stored in its container.

Sample Analysis

Test samples were prepared for analysis by Soxhlet extraction in ether:n-hexane (6:94) for 16 hours followed by a Florisil cleanup. A surrogate compound (p terphenyl-d14) was added to each sample as a QC check on the process.

Analysis was performed with a Fisons VG MD800 GC/MS instrument in selected ion monitoring mode. A DB-5 column, 0.25-mm-i.d. - x 30-m, was used for the analysis. Quantitation was based on a five-point standard calibration curve, and continuing calibration was performed by using the mid level standard.

Results and Discussion

The analytical results for blanks, field spikes, surrogate recoveries, and GC/MS calibrations were acceptable for all seven sets of test samples analyzed. Pesticide formula applications were uniform with the results of deposition coupon analysis yielding an average of 23% RSD for three replicates per test.

The results for method sampling precision, shown in Table 1, indicate that all three methods achieved relatively good precision. From a total of 63 sets of triplicate results (7 tests x 3 methods x 3 pesticides), the overall average sampling precision was 31.3% RSD (range = 2.4 86.1% RSD). The Dow sled achieved the best sampling precision with an average of 25.4% RSD, followed by the California roller method at 30.7%, and the PUF roller at 37.9%. Using the calculated average deposition rate for each test, the mean transfer efficiency rate was determined for the three replicate samples collected using each test method. The average results for the seven tests performed in the study are shown in Table 2. The results indicate that the transfer efficiency for each method is generally quite consistent for all three target pesticides and over the whole range of subject operators. The California roller produced the highest transfer efficiency results, with an overall average of 5.0%. The rates for the PUF roller and Dow sled methods were similar, with the Dow sled showing only slightly higher values on average. The PUF roller averaged a 1.7% tranfer efficiency rate, while the Dow sled averaged 2.1%. Since both of these test methods were designed to mimic the exposure of a 20-lb child to surface residues, it is not surprising that the transfer efficiency results are similar. The California roller method seemed to be subject to problems during execution of the technique by several operators. On many occasions the dosimeter was observed to move or slide on the carpet surface during the rolling operation. This problem might account for higher transfer efficiency results for some of the test samples collected relative to tests during which the dosimeter remained stationary during the rolling operation.

Table 1. Round-Robin Test Results Summary - Sampling Precision*

	Chlorpyrifos	Pyrethrin I	Piperonyl Butoxide	Average
PUF Roller	28.3%	45.7%	39.7%	0.379
California Roller	27.1%	35.2%	29.7%	0.307
Dow Drag Sled	23.5%	26.8%	25.8%	0.254

* Mean % RSD for 3 replicates per test, average results for 7 tests.

Table 2. Round-Robin Test Results Summary - Transfer Efficiency*

	Chlorpyrifos	Pyrethrin I	Piperonyl Butoxide	Average
PUF Roller	1.4%	1.9%	1.8%	1.7%
California Roller	4.2%	4.2%	6.6%	5.0%
Dow Drag Sled	1.9%	2.1%	2.3%	2.1%

* Mean % transfer for 3 replicates per test, average results for 7 tests.

The results of this study were compared to those of the study that led to the recommendation for this round robin test. The results for transfer rates of the three test methods were similar in both studies for tests on new carpets. In the earlier study, however, tests performed on carpets that were used and then repeatedly cleaned by chemical treatment yielded transfer rates that were ten times lower than those from tests on new carpets.

The volunteers in the round robin study provided a written evaluation of all three test methods following completion of the testing. The methods were rated numerically as to (a) ease of use, (b) convenience, (c) transporting apparatus in the field, and (d) ease of handling the sample media. Responses were also obtained pertaining to the time requirements and simplicity of each method. The California roller method was consistently rated lower in every catergory, while the PUF roller and Dow sled methods were generally rated highly and about equal to one another.

Conclusions and Recommendations

1. The volunteers were readily able to perform the dislodgeable residue sampling procedures with only minimal prior orientation and instruction.
2. The pesticide application procedure used for each of the seven round-robin tests yielded uniform results. The average deposition rate (mean±SD, $\mu g/cm^2$) was 17.8±4.1 for chlorpyrifos, 1.33±0.61 for pyrethrin I, and 10.1±2.3 for piperonyl butoxide.
3. One of the primary objectives of this study was to determine the level of sampling precision that could be attained with each of the test methods when used by typical operators under controlled conditions. As measured by the calculated %RSD for three replicate samples, the Dow sled method registered the highest overall sampling precision (25.4% RSD). The PUF roller method registered the lowest sampling precision (37.9% RSD), and results for the California roller method, modified to include use of a custom handle assembly, were slightly better than the average (30.7% RSD).
4. The other main objective criterion measured by the results from the round-robin testing was that of method transfer efficiency. Transfer efficiency is defined as the ratio of a method's transfer rate ($\mu g/cm^2$)

to the deposition rate (μg/cm^2) as determined by the mean value for three deposition coupons. The mean transfer efficiency for the California roller was high (5.0%), while the PUF roller and the Dow sled mean transfer efficiencies were nearly identical at 1.7% and 2.1%, respectively.

5. Transfer efficiency for all three dislodgeable residue test methods was higher by about one order of magnitude in the round robin tests on new carpets as compared to that determined in similar experiments performed on used carpets in an earlier study of this type. Method transfer rate, the ratio of the amount (μg) of pesticide collected to the area (cm^2) of carpet surface traversed by the sampler, may be profoundly influenced, as the earlier work suggests, by the type, age, and prior surface treatment of the carpets being tested.

6. The results of subjective evaluations of the three test methods by the six round-robin test volunteers indicated a clear consensus in rating the California roller method lower overall as compared to the PUF roller and Dow sled methods. The latter two methods were rated highly and almost equal to one another according to the volunteers' responses to criteria addressed in the opinion survey.

7. The quantitative results of the study along with the subjective evaluations by the volunteers lead to the conclusion that the California roller method is the least suitable of the three test methods for sampling dislodgeable residue on carpets. Its high transfer efficiency (5.0%) is less representative of actual human skin transfer efficiency, and the method involves bulky equipment that is difficult to use. Both the PUF roller and the Dow drag sled methods performed well and are considered equally suitable.

8. Although the results of this study show that reproducible and consistent data can be obtained for dislodgeable pesticide residues on new carpet by using any of the three test methods under evaluation, the substantial difference in measured transfer efficiency rates between this study and one conducted earlier are cause for concern. We therefore recommend that tests be conducted with one or more of the residue sampling methods on both new and used carpets of varying types, including carpets that have, and have not, been chemically treated.

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Christopher R. Fortune is with ManTech Environmental Technology, Inc., Research Triangle Park, NC 27709
***Robert G. Lewis** is the EPA Project Officer (see below).*
The complete report, entitled "Round-Robin Testing of Methods for Collecting Dislodgeable Residues from Carpets," (Order No. PB98-117989; Cost: $21.50, subject to change) will be available only from:
National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650
The EPA Project Officer can be contacted at:
National Exposure Research Laboratory
U.S. Environmental Protection Agency
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